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DETERMINATION OF IRON IN CHROMIUM PLATING AND POLISHING SOLUTIONS BY ATOMIC ABSORPTION SPECTROMETRY

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The chemical literature lacks a specific analytical method for adequately monitoring iron in chromium plating and polishing solutions during the plating and polishing processes. In this report, a specific method is presented for analyzing and monitoring iron during these processes. The optimum operating range of the iron is generally around 10 g/l maximum in both the chromium plating and the polishing solutions. The resulting precisions are in the range of 0.5 to 1.5 g/l, providing adequate monitoring of these solutions supported by six years of testing.

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INTRODUCTION

Iron is produced as an unwanted by-product of polishing and chromium plating solutions for lcw alloy steels (refs 1-3). The chemical literature lacks a specific analytical method for adequately monitoring iron in chromium plating and polishing solutions during the plating and polishing processes. Lack of optimization of these plating and polishing solutions causes serious problems for the chromium plating industry such as poor quality products, wasted human resources, and wasted electrical energy.

A common chemical analysis method to determine iron in the presence of chromium plating and polishing solutions is composed of an alkaline precipitation of iron (ref 4). This method provides adequate precisions, but an unacceptable analysis time of two days.

The specific method given in this report provides both acceptable analysis and monitoring of iron in chromium plating and polishing solutions. This method consists of atomic absorption (AA) spectrometry (ref 5).

EXPERIMENTAL PROCEDURE

Strict analytical chemistry methods and procedures are followed throughout this experimental procedure section. An excellent source of reference for these methods and procedures is by Fritz and Schenk (ref 4).

One analytical reagent grade standard solution is required. It is a 1.000 ± 0.005-g/l iron solution also containing 50 milliliters (ml) of concentrated nitric acid per liter that meets American Chemical Society (ACS) and American Society For Testing and Materials (ASTM) Standards (ref 6).

References are listed at the end of this report.

Tables II and III present the sample solution data for iron in chromium plating and polishing solutions, respectively. These sample solutions are diluted 1:2000 in order to attain detector linearity. Due to a linear operating range, the following simplified calculation is used to determine iron concentration in the original chromium plating and polishing sample solutions:

g/l Iron = (10)(sample absorbance/5 ppm standard absorbance)

The chromium plating sample solution in Table II has a 2.72-g/l iron concentration, and the polishing sample solution in Table III has a 5.44-g/l iron concentration.

Chromium plating solutions typically contain 240 to 260 g/l chromic acid and 2.4 to 3.0 g/l sulfuric acid, while polishing solutions typically contain 640 to 730 g/l phosphoric acid and 795 to 895 g/l sulfuric acid. The major components of these sample solutions do not interfere with the iron determination by AA spectrometry (ref 6).

It is useful to evaluate the variations in precision for the materials and methods used. Tables IV through VIII present these data for the 0.500-ml micropipets, 0.250-ml micropipets, 0.050-ml micropipets, the 100-ml class-A volumetric flasks, and the 1-g/l iron standard solution, respectively. Variations in precision are also evaluated for the AA spectrometer. Table IX presents these data for six consecutive replicates of the 5-ppm iron standard solution.

The data obtained by this specific method are sufficient to adequately monitor the iron in chromium plating and polishing processes, thus providing efficient use of resources. The optimum operating range of iron is generally around 10 g/l maximum, and the resulting precisions are in the range of 0.5 to 1.5 g/l, providing adequate monitoring of these solutions supported by six years of testing.

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4. J. Fritz and G. Schenk, Quantitative Analytical Chemistry, Fifth Edition, Allyn and Bacon, Inc., Boston, MA, 1987.
5. H. Bauer, G. Christian, and J. O'Reilly, Instrumental Analysis, Allyn and Bacon, Inc., Boston, MA, 1978.
6. "AA/ICP Operators Manual," Perkin-Elmer Corp., Norwalk, CT, 1981.

TABLE I. STANDARD SOLUTION DATA FOR IRON

Replicate	Absor. (AU) 0.00 ppm Iron	Absor. (AU) 2.50 ppm Iron	Absor. (AU) 5.00 ppm Iron
1	0.003	0.100	0.197
2	0.002	0.104	0.200
3	0.002	0.098	0.202
X(avg)	0.002	0.101	0.200

TABLE II. EXPERIMENTAL SAMPLE SOLUTION DATA FOR IRON IN CHROMIUM PLATING SOLUTIONS

Replicate	Sample Iron Absor. (AU)	Sample Iron Conc. (ppm)
1	0.052	1.30
2	0.055	1.38
3	0.056	1.40
X(avg)	0.054	1.36

TABLE III. EXPERIMENTAL SAMPLE SOLUTION DATA FOR IRON IN POLISHING SOLUTIONS

Replicate	Sample Iron Absor. (AU)	Sample Iron Conc. (ppm)
1	0.112	2.80
2	0.108	2.70
3	0.106	2.65
X(avg)	0.109	2.72

TABLE IV. PRECISION OF MICROPIPETTING 0.500 ml

Replicate	Volume (ml)*
1	0.5026
2	0.5115
3	0.5118
4	0.5013
5	0.5054
6	0.5079
X(avg)	0.5068
Sn	0.0044

*Volumes are calculated from the weight-volume relationship of each micropipetted deionized water solution corrected for temperature.

TABLE V. PRECISION OF MICROPIPETTING 0.250 ml

Replicate	Volume (ml)*
1	0.2582
2	0.2497
3	0.2546
4	0.2545
5	0.2557
6	0.2532
X(avg)	0.2543
Sn	0.0028

*Volumes are calculated from the weight-volume relationship of each micropipetted deionized water solution corrected for temperature.

TABLE VI. PRECISION OF MICROPIPETTING 0.050 ml

Replicate	Volume (ml)*
1	0.0494
2	0.0508
3	0.0533
4	0.0522
5	0.0497
6	0.0517
X(avg)	0.0512
Sn	0.0015

*Volumes are calculated from the weight-volume relationship of each micropipetted deionized water solution corrected for temperature.

TABLE VII. PRECISION OF A 100-ml CLASS-A VOLUMETRIC FLASK

Replicate	Volume (ml)*
1	100.14
2	99.97
3	99.89
4	100.12
5	100.03
6	100.06
X(avg)	100.04
Sn	0.09

*Volumes are calculated from the weight-volume relationship of the contained deionized water solution corrected for temperature.

TABLE VIII. PRECISION OF A 1-g/l IRON STANDARD SOLUTION

Replicate	Iron Conc. (g/l)*
1	1.003
2	1.000
3	1.002
4	1.001
5	1.002
6	1.002
X(avg)	1.002
Sn	0.001

*Iron concentrations are determined by the alkaline precipitation method in Fritz and Schenk (ref 4) which is a standard chemical analysis method for iron.

TABLE IX. PRECISION OF A 5-ppm IRON STANDARD SOLUTION BY AA SPECTROMETRY

Replicate	Absor. (AU) 5.00 ppm Iron
1	0.201
2	0.198
3	0.203
4	0.200
5	0.200
6	0.204
X(avg)	0.201
Sn	0.002

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